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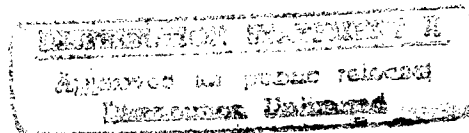
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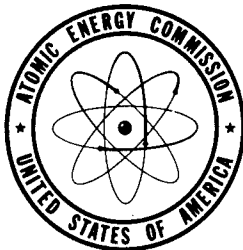
ORGANIC PRECIPITANTS AND  
COMPLEXING AGENTS FOR URANIUM

By  
E. J. Kahler  
G. W. Kinzer  
G. A. Lutz  
R. H. Poirier  
A. E. Bearse



June 30, 1948

Battelle Memorial Institute  
Columbus, Ohio



Technical Information Extension, Oak Ridge, Tennessee

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## INTRODUCTION

This project is concerned with a fundamental study of methods of recovering uranium from dilute aqueous solutions by means of organic precipitants or complexing agents. In many instances, solutions of this type are encountered as effluents from processing plants or as leach solutions from low-grade uranium-bearing ores, from which it is desired to obtain the uranium in concentrated form without using excessive amount of neutralizing agents. Many of these solutions contain large amounts of extraneous ions, which in some cases may be present to the extent of several hundred times the quantity of uranium in the solution. Therefore, the effect of interfering ions on uranium extraction is being investigated.

Work on this project was initiated in September, 1947, but due to the delay in obtaining clearance on personnel, several months elapsed before a satisfactory rate of effort could be achieved. Considerable time has been spent in reviewing the literature on organic precipitants as related to this project. This review included the published literature and classified reports furnished by the Atomic Energy Commission.

Initial work was carried out on nearly neutral solutions but later work was concentrated on solutions containing mineral acids or sodium carbonate or bicarbonate, since the latter types of solutions are more typical of those encountered in practice. One phase of the work involved testing of a variety of organic compounds as precipitants, while a second phase was concerned with the study of organic compounds capable of forming uranium complexes which might be soluble in organic solvents. Numerous

organic compounds were synthesized during the course of this work.

The period covered by this report is from September, 1947, to June 30, 1948.

### SUMMARY

Experiments carried out during the early stages of this project demonstrated that many types of organic compounds are capable of precipitating uranium almost quantitatively from a nearly neutral uranyl nitrate solution devoid of interfering ions. Unfortunately, this type of solution is seldom encountered in practice, and attempts to apply the same organic compounds to acid solutions or sodium carbonate solutions were much less successful.

Prior to the start of this project, it was found on another project at Battelle that long-chain amines were effective in precipitating uranium from superphosphate leach solutions. Since this appeared to be a novel type of precipitant, considerable attention has been given to a more fundamental investigation of these amines. A commercial product (Armeen CD) was carefully fractionated to give pure amines corresponding to C<sub>10</sub>, C<sub>12</sub>, and C<sub>14</sub>. It was found that these pure compounds were definitely inferior as precipitants to the crude mixture of amines or to the intermediate cuts obtained during the fractionation. Furthermore, the pure amines were not so effective as synthetic mixtures of the pure amines. In an extension of this work, a series of amines containing odd numbers of carbon atoms from C<sub>9</sub> to C<sub>19</sub> was synthesized and tested. The same phenomenon

was observed. For example, the pure C<sub>9</sub> and C<sub>11</sub> amines precipitated only 20 to 30 per cent of the uranium from a water extract of superphosphate; whereas, the same weight of a mixture of equal parts of the two amines precipitated about 85 per cent of the uranium under the same conditions. These results have not yet been explained.

In addition to the work on precipitation with organic materials, considerable attention has been given to studies on the formation of complexes of uranium with organic substances and subsequent removal of these complexes from the aqueous phase into an organic solvent. Cupferron, 8-hydroxyquinoline, and the sodium dialkyl dithiocarbamates are examples of this type of organic substance, which were found to give good removal of uranium from uranyl nitrate solutions. However, application to more complex solutions containing sodium carbonate or mineral acids other than nitric acid was considerably less promising.

Numerous organic compounds were tested for their ability to form solvent-extractable complexes with uranium in superphosphate solutions. Nearly all of these were too ineffective to be of interest, but the use of phenyl biguanide hydrochloride with isopropyl acetate as a solvent and 2,4-diaminodiphenyl amine with methyl isobutyl ketone as a solvent gave about 50 per cent removal of the uranium from the phosphate solutions.

Results on the removal of uranium from sodium carbonate or bicarbonate solutions by the use of organic reagents as precipitants or complexing agents have been very unpromising. Apparently, the uranium in the sodium uranyl tricarboxylate complex is too firmly bound to be available for combination with the organic addition agent.

Results in the case of test solutions containing considerable sulphuric acid were also very unsatisfactory. In these solutions, it is probable that the organic uranium compound would be decomposed as rapidly as it is formed and, therefore, would not exist in sufficient concentration to be effective. Additional work with more dilute sulphuric acid solutions is underway, since it is anticipated that the organic complexes will be more stable in such solutions.

A brief bibliography of some of the more pertinent references studied in connection with this project on organic precipitants and complexing agents for uranium is included in this report.

#### DESCRIPTION OF TEST SOLUTIONS

Solutions containing 12.5 mg. of uranium per liter as uranyl nitrate were prepared for evaluation of organic precipitants or complexing agents for the removal of uranium. The compositions were representative of leach solutions obtained in the extraction of uranium from phosphate rock or from other low-grade materials (Table 1). The pH of these solutions varied from zero for the sulphuric acid solutions to approximately eleven for the sodium carbonate solutions.

In preparing synthetic superphosphate solutions, primary calcium phosphate was dissolved in dilute phosphoric acid and separate solutions of sodium fluoride, aluminum sulphate, ferric sulphate, sulphuric acid, and uranyl nitrate were then added.



TABLE 1. COMPOSITION OF SOLUTIONS

Solutions	pH	Ions Present (Milliequivalents/Liter)												
		Al <sup>+++</sup>	Ca <sup>++</sup>	Fe <sup>++</sup>	Mg <sup>++</sup>	Na <sup>+</sup>	Si	U	HCO <sub>3</sub> <sup>-</sup>	CO <sub>2</sub>	F <sup>-</sup>	PO <sub>4</sub> <sup>=</sup>	SiO <sub>3</sub> <sup>=</sup>	SO <sub>4</sub> <sup>=</sup>
Superphosphate (Phosphoric acid)	2.1*	14.7	312.5	1.06	-	-	-	0.0522	-	-	7.9	679.8	-	23.0
Sulphuric acid	0	108.8	-	87.5	20.5	-	-	0.0522	-	-	-	31.2	-	2000
Sodium bi- carbonate	8.3	0.83	-	0.89	-	1120	-	0.0522	1081	-	-	-	2.40	15.1
Sodium carbonate	11.0	1.86	-	0.25	-	-	0.16	0.0522	-	1420	-	0.30	-	36.0

\* Synthetic superphosphate solutions had a slightly lower pH than the corresponding leach solutions (2.1 against 2.7).

## DETAILS AND DISCUSSION OF RESULTS

During the early course of this work, attention was given to the removal of uranium from practically neutral uranyl nitrate solutions which did not contain any interfering ions. It was soon realized that the effectiveness attained in the removal of uranium from such solutions could not be expected in the case of acid or carbonate leach solutions. Preliminary observations and also more recent observations with more complex solutions are discussed in this report.

### PRECIPITATION OF URANIUM FROM URANYL NITRATE SOLUTIONS

(Table 2, Appendix)

Aniline diacetic acid, diacetoacetyl ethylene diamine, diglycol stearate, ditolylguanidine, and 3-methyl-1-phenyl-5-pyrazolone were very effective in removing small amounts (10 mg. per liter) of uranium from uranyl nitrate solutions. Large amounts (10 g.) of reagents were used in preliminary screening tests. Diglycol stearate in a concentration as low as 0.2 g. per 500 cc. removed uranium quantitatively from a neutral nitrate solution containing 5 mg. of uranium.

Lauric acid and also stearic acid (1 g. per liter) precipitated 90 and 96 per cent, respectively, of the uranium from a nitrate solution at a pH of 5.0.

Only small amounts (0.25 per cent) of sodium bicarbonate or ammonium carbonate could be tolerated in the precipitation of uranium from uranyl nitrate solutions using long-chain amines (Armeen CD) as precipitants

PRECIPITATION OF URANIUM FROM SUPERPHOSPHATE SOLUTIONS.  
EVALUATION OF LONG-CHAIN AMINES.

(Figures 2, 3, and 4, Appendix)

The effectiveness of Armeen CD\*, a mixture of even carbon atom aliphatic amines distilling mostly between 210 and 255°C. (410 and 480°F.), as a precipitant for uranium from solutions obtained by water extraction of superphosphate fertilizer was observed by Igelsrud, Topical Report November 15, 1947 (Contract No. W-38-094-Eng-27). The results reported in the present study cannot be compared directly with those of Igelsrud because of a difference in the amounts of amine used and in the pH of the solutions.

In the current study, Armeen CD was fractionated, and the constituents were evaluated as precipitants in phosphate solutions. Rectification of Armeen CD is represented in Figure 1, Appendix. In general, fractions which were collected over a wide range of temperature compared favorably in precipitating qualities with crude Armeen CD. However, pure amines, e.g., C<sub>10</sub>, C<sub>12</sub>, and C<sub>14</sub> obtained in the fractionation were significantly inferior to broad fractions of amines or to the crude Armeen CD. These observations are illustrated in Figures 2 and 3. Using crude Armeen CD in an amount of 5 grams per 400 cc. of superphosphate solution containing 5 mg. of uranium, 53 per cent of the uranium was precipitated. When Armeen CD was fractionated and pure fractions and also intermediates were evaluated as precipitants, results illustrated in Figure 2 were

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\* Armour and Company.

obtained. In all of the work described in this section and represented in Figures 2, 3, and 4, five grams of amine was added to 400 cc. of the phosphate solution containing 5 mg. of uranium. Composition of the phosphate solution is given in Table 1. Using  $C_{10}$ ,  $C_{12}$ , and  $C_{14}$  fractions, 10 to 13 per cent of the uranium in solution was precipitated. However, use of intermediate, wide-boiling fractions gave a minimum of 40 per cent ( $C_{13}$  range) and a maximum of 80 per cent ( $C_{16}$  range).

Experiments in which binary mixtures of adjacent long-chain amines containing an even and odd number of carbon atoms from nonyl to nonodecyl were compared in the same proportions with the individual amines, illustrate the superiority of amine mixtures (Figures 3 and 4). In all cases, a decidedly greater portion of uranium was precipitated when mixtures of amines were used than when pure amines were tested. Since mixtures of the low molecular weight amines are more effective than mixtures of the high molecular weight amines when compared on a weight basis, it is believed that the amino group is a critical factor in these precipitations. However, an adequate explanation of the difference in precipitating properties between pure fractions and mixtures is not available at present. A study of the physical properties of amines, especially mixtures of amines, might provide the answer. Work along this line is in progress.

#### COMPLEXING AND SOLVENT EXTRACTION

Since the concentration of uranium in the solutions being investigated is approximately 12.5 mg./liter, extremely insoluble precipitants are required if quantitative recovery of uranium is to be realized. In

many cases, the solubility of the complex may not be exceeded; and, consequently, precipitation cannot be detected, although a complex may exist between the organic reagent and the uranium ions. Provided that this complex is soluble in organic solvents and is reasonably stable in the aqueous system being investigated, it should be possible to recover uranium from dilute aqueous solutions by addition of a complexing agent followed by extraction with a suitable solvent. Numerous types of organic complexing agents were tried in conjunction with representative solvents on different types of solutions as described in the following sections.

#### URANYL NITRATE SOLUTIONS

(Table 3, Appendix)

Extraction of a uranyl nitrate solution (5 mg. uranium/400 cc.) with 50 cc. of tricresyl phosphate gave 96 per cent removal of uranium.

Experiments in which 1.0 g. of Armeen CD was employed to complex the uranium for extraction with methyl isobutyl ketone gave 78 per cent removal.

When 1.0 g. of sodium thiocyanate was added to 400 cc. of the nitrate solution prior to extraction, 98 per cent recovery was realized. The extraction decreased to 86 per cent when 10 g. of sodium thiocyanate was used.

Addition of 0.2 g. of sodium dibutyl dithiocarbamate to 400 cc. of a uranyl nitrate solution followed by extraction with 50 cc. of amyl acetate or amyl alcohol gave 76 to 98 per cent removal of the uranium. Maximum removal was obtained when the pH was adjusted to 2.9 with nitric acid.

# SUPERPHOSPHATE SOLUTIONS

(Tables 4 and 5, Appendix)

Numerous types of organic compounds were examined as reagents which might be capable of forming solvent-extractable complexes with uranium in superphosphate solutions. On the basis of these preliminary studies, only a few of the compounds tested gave promising results:

Complexing Agent	Grams Added per 400 cc. of Solution Containing 5 mg. of Uranium	Solvent	Volume of Solvent, cc.	Uranium Extracted, Per Cent
Phenylbiguanide hydrochloride	0.2	Isopropyl acetate	150	55
2,4-diamino diphenyl amine	0.6	Methyl isobutyl ketone	150	49
o-Aminophenol	0.5	Ditto	150	36
Armeen CD	0.5	"	150	34

It is interesting to note that compounds which gave moderate removal of uranium contained at least one amino group. In consequence of this, special attention will be given to derivatives of primary amines, and to secondary and tertiary amines.

# SULPHURIC ACID SOLUTIONS

(Table 6, Appendix)

Only a few compounds have been evaluated for complexing and subsequent extraction of uranium from sulphuric acid solutions containing a

high concentration of interfering ions. The sodium salts of several alkyl and aryl dithiocarbamates, as well as Armeen CD, did not form complexes which were readily extractable with amyl acetate. Less than 10 per cent recovery resulted.

A study of sulphuric acid solutions containing only uranium salts might give screening information essential in the examination of more complex solutions.

#### SODIUM BICARBONATE SOLUTIONS

(Table 7, Appendix)

The sodium salts of several alkyl and aryl thiocarbamates were ineffective as complexing agents in bicarbonate solutions. Extraction with amyl acetate gave very little removal of uranium. Armeen CD and phenyl biguanide hydrochloride also were unsatisfactory.

#### SODIUM CARBONATE SOLUTIONS

(Table 8, Appendix)

The potassium salts of several xanthates along with methyl isobutyl ketone as a solvent for extraction, were examined briefly in the removal of uranium from carbonate solutions. The use of the ethyl xanthate salt gave 21 per cent extraction. Other xanthates ranged from 8 to 12 per cent. Carbon tetrachloride, nitrobenzene, aniline, diamyl sulfide, trichloroethylene, dibutyl cellosolve, chloroform, and benzene were ineffective in extracting uranium from similar solutions.

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### PLANS FOR FUTURE WORK

In view of the significance of pH on the effectiveness of organic precipitants and complexing agents, it is planned to study this factor more carefully for each organic reagent tested. This will be done by evaluating reagents in buffered solutions at a series of pH values in order to determine those reagents which are effective over a wide range. The more promising compounds will then be studied more carefully to establish the effects of interfering ions.



Since it has been suggested that tetravalent uranium may be easier to precipitate or complex than the uranyl ion, studies are to be carried out on the former. All work to date has been concerned with hexavalent uranium.

**APPENDIX**

## APPENDIX

### PREPARATION OF REAGENTS

Reagents which were not available commercially were prepared according to procedures described in the literature.

#### AMINES

Odd and even carbon atom amines were prepared by dissolving the corresponding nitrile in methanol plus liquid ammonia and hydrogenating at 125°C. (260°F.) and 2500 to 3500 p.s.i. pressure in presence of Raney nickel catalyst. The nitriles were obtained by refluxing an ethanolic solution of potassium cyanide and the proper alkyl bromide.

#### ETHYLENE DIAMINE-ALDEHYDE OR KETONE CONDENSATION PRODUCTS

These compounds were prepared according to the procedure of Mason, Ber., 20, 267-277 (1887).

#### THIOCARBAMATES

The sodium salts of alkyl, aryl, and alkylaryl thiocarbamates were prepared from the appropriate amine, excess carbon disulphide and 50 per cent aqueous sodium hydroxide solution.

#### XANTHATES

Xanthates were prepared by the slow addition of the appropriate alcohol to a mixture of carbon disulphide and powdered potassium hydroxide.

## PROCEDURES USED IN EVALUATING REAGENTS

### Precipitation Tests

In general, the reagent to be tested was added to 400 cc. of stock solution containing 5 mg. of uranium, with or without interfering ions. In the case of the uranyl nitrate solutions, small amounts of mineral acids or alkali were introduced to obtain the indicated acidity. Solutions were stirred one hour, filtered, and the filtrates were analyzed for uranium.

### Complexing and Solvent-Extraction Tests

Complexing agents (0.2 to 0.6 g.) were added to 400 cc. of stock solution and the mixture was stirred fifteen minutes after the desired solvent had been added. In the early phase of the work, triple additions and triple extractions were carried out. More recently, it was found that single processes were satisfactory for screening tests.

TABLE 2. PRECIPITATION OF URANIUM FROM URANIL NITRATE SOLUTIONS  
(Solutions Contained 5 mg. U per 400 cc.)

Reagent	Grams of Reagent per 400 cc. of Solution	pH	Uranium Removed, Per Cent
Aniline diacetic acid	10	2.8	98
Diacetoacetylene diamine	10	2.8	98
Diglycol stearate	10	5.5	99
Diglycol stearate	10	7.0	99
Diglycol stearate	0.2	7.0	99
Diglycol stearate	10	2.9	12
Diglycol stearate	10	9.0	16
Ditolylguanidine	10	7.3	92
Lauric acid	1.0	5.0	90
3-Methyl-1-phenyl-5-pyrazolone	10	3.5	76
Stearic acid	1.0	5.0	96

TABLE 3. COMPLEXING AND SOLVENT EXTRACTION OF URANIUM  
FROM URANYL NITRATE SOLUTIONS

(Solutions Contained 5 mg. of U per 400 cc.)

Complexing Compound	Grams per 400 cc. of Solution	pH	Solvent	Volume of Solvent, cc.	Uranium Extracted, Per Cent
"Cupferron"	3 x 1	3.0	Methyl iso- butyl ketone	3 x 50	98
8-Hydroxyquinoline sulfate	2 x 2	8.0	Amyl acetate	2 x 50	98
Sodium allyl xanthate	3 x 0.2	8.0	Amyl acetate	3 x 50	98
Sodium dibutyldithiocarbamate	3 x 0.2	2.9	Amyl acetate	3 x 50	98
Sodium dibutyldithiocarbamate	3 x 0.2	8.7	Amyl alcohol	3 x 50	76
Sodium dibutyldithiocarbamate	3 x 0.2	2.9	Amyl alcohol	3 x 50	98
Sodium dibutyldithiocarbamate	3 x .02	8.7	Amyl alcohol	3 x 50	80
None	-	3.0	Tricresyl phosphate	50	96

TABLE 4. SCREENING TESTS OF COMPLEXING AGENTS FOR EXTRACTION OF URANIUM FROM SUPERPHOSPHATE SOLUTIONS WITH METHYL ISOBUTYL KETONE (1)

Complexing Agent	Uranium Extracted, Per Cent
None	0
Acetonyl acetone	5
Acetyl acetone	15
o-Aminophenol	36
Anthranilic acid	19
Benzil (dibenzoyl)	10
$\alpha$ -Benzoin oxime	6
Catechol	21
Diacetyl	10
Diacetylacetonyl ethylene diimine	0
Diacetyl dihydrazone	0
2,4-Diaminodiphenylamine	49
Dibenzyl ethylene diimine	2
Di-(o-chlorobenzyl-) ethylene diimine	0
Di-cinnamyl ethylene diimine	15
Di-(2,3-dimethoxybenzyl) ethylene diimine	2
Di-(2-hydroxy-3-methoxybenzyl) ethylene diimine	6
Di-isopropyl dixanthogen	13
Di-methylamine-methylphenol	23

TABLE 4. (CONTINUED)

Complexing Agent	Uranium Extracted, Per Cent
o-Di-methylamine-p-octylphenol	19
Di- $\alpha$ -methylbenzyl ethylene diimine	6
s-Diphenyl guanidine	6
s-Diphenyl thiourea	2
Dithiobiuret	0
Di-o-tolylguanidine	13
s-Di-o-tolylguanidine	0
s-Di-o-tolyl thiourea	12
s-Di-o-tolyl urea	20
Ethylene thiourea	19
Guanidine carbonate	4
Guanidine hydrochloride	14
Guanidine nitrate	6
Guanidine sulfate	10
Guanyl urea sulfate	14
2-Hydroxyquinoline	19
8-Hydroxyquinoline	23
8-Hydroxyquinoline sulfate	15
8-Hydroxyquinoline-5-sulfonic acid	17



TABLE 4. (CONTINUED)

Complexing Agent	Uranium Extracted, Per Cent
2-Mercapto-4,6,6-trimethyl thiazine	19
Mixed aliphatic thiazyl disulfides	2
Mixed ethyl and dimethyl mercaptothiazoles	17
o-Phenylenediamine	25
Phthalic acid	15
Quinaldine	19
Quinoline	21
Rhodanine	13
Salicylaldoxime	8
Salicylic acid	28
Stearic acid	21
Thenoylfluoroacetone	8
Thiosalicylic acid	19

(1) 400 cc. of superphosphate solution (See Table 1) treated with 3 x 0.2 gram of complexing agent and extracted with 3 x 50 cc. of methyl isobutyl ketone.

TABLE 5. SOLVENT STUDY OF MOST PROMISING COMPLEXING AGENTS  
ON SUPERPHOSPHATE SOLUTIONS (1)

Complexing Agent	Solvent	Uranium Extracted, %
Phenylbiguanide hydrochloride	Isopropyl acetate	55
	Amyl acetate	11
	Methyl ethyl ketone	40
	Methyl isobutyl ketone	0
	Ethyl ether	8
	Ethylene dichloride	9
	Benzene	0
Armeen CD	Methyl isobutyl ketone	28
	Amyl acetate	30
	Benzene	30
	Ethylene dichloride	34
	None (filtered ppt.)	21
2,4-Diaminodiphenylamine	Methyl isobutyl ketone	36
	Amyl acetate	10
	Benzene	12
	Ethylene dichloride	12
	Chloroform	20

(1) 400 cc. of superphosphate (see Table 1) treated with 3 x 0.2 gram of complexing agent and extracted with 3 x 50 cc. of solvent.

TABLE 6. COMPLEXING AND SOLVENT EXTRACTION OF  
URANIUM FROM SULPHURIC ACID SOLUTIONS(1)

Complexing Agent	Solvent	Uranium Extracted, Per Cent
Armeen CD	Amyl Acetate	6
Sodium dibenzylldithiocarbamate	Ditto	2
Sodium dibutylldithiocarbamate	"	8
Sodium dicyclohexylldithiocarbamate	"	4
Sodium diethylldithiocarbamate	"	8

(1) 400 cc. of sulphuric acid solution (see Table 1) treated with 3 x 0.2 gram of complexing agent and extracted with 3 x 50 cc. of amyl acetate.

TABLE 7. COMPLEXING AND SOLVENT EXTRACTION OF URANIUM  
FROM SODIUM BICARBONATE SOLUTIONS(1)

Complexing Agent (Sodium Salt of Dithiocarbamates)	Solvent	Uranium Extracted, Per Cent
Diethyl	Amyl acetate	8
Lauryl	Ditto	2
Phenyl ethyl	"	0

(1) 400 cc. of sodium bicarbonate solution (see Table 1) treated with 3 x 0.2 gram of complexing agent and extracted with 3 x 50 cc. of amyl acetate.

TABLE 8. COMPLEXING AND SOLVENT EXTRACTION OF URANIUM  
FROM SODIUM CARBONATE SOLUTIONS(1)

Complexing Agent (Potassium Salt of Xanthate)	Solvent	Uranium Extracted, Per Cent
Ethyl	Methyl isobutyl ketone	21
Ethylene glycol	Ditto	12
Isopropyl	"	12
3-Methylcyclopentyl	"	8

(1) 400 cc. of sodium carbonate solution (see Table 1) treated with 3 x 0.2 gram of complexing agent and extracted with 3 x 50 cc. of methyl isobutyl ketone.

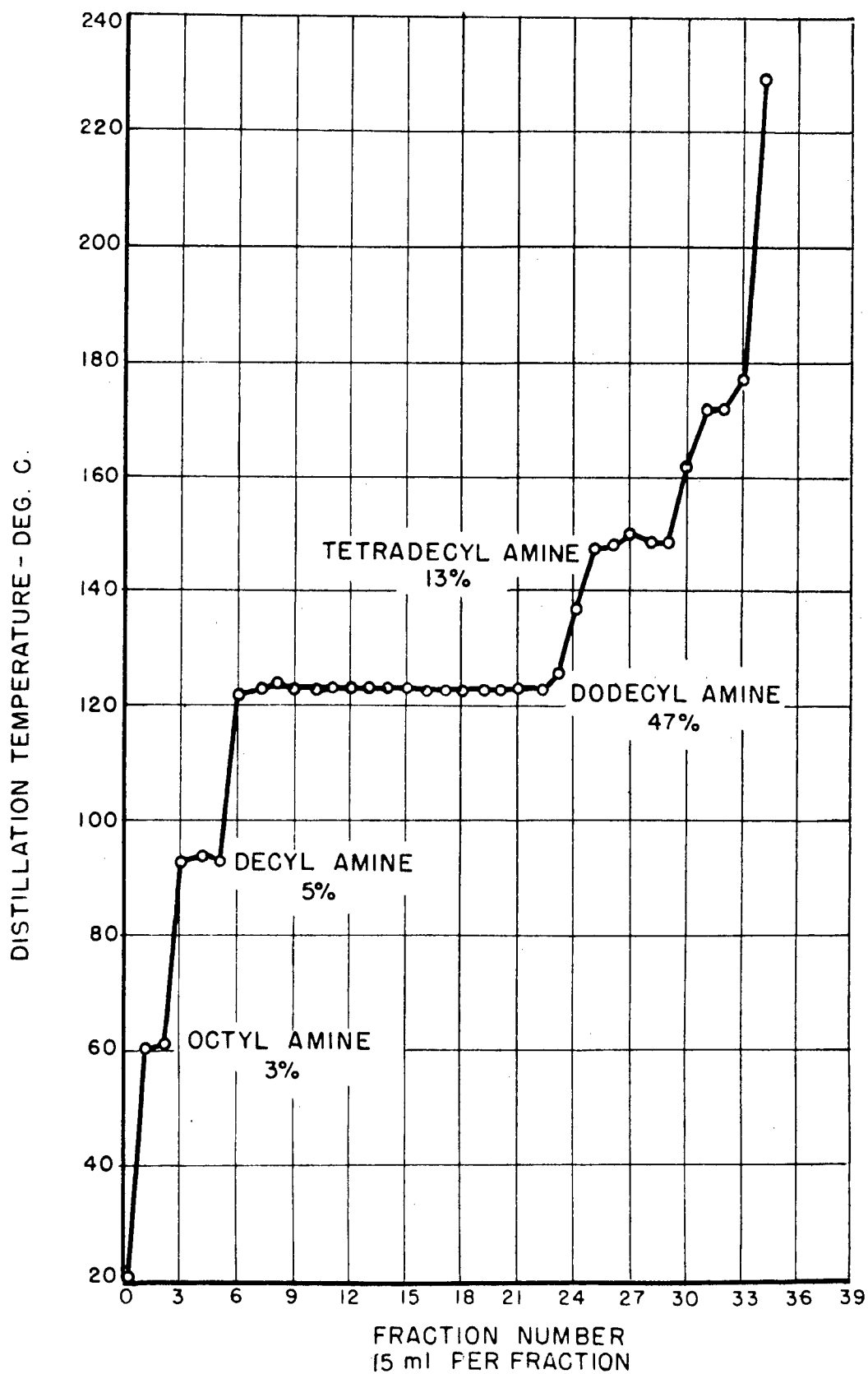


FIGURE I - RECTIFICATION OF ARMEEN CD AT 7-10 m.m.

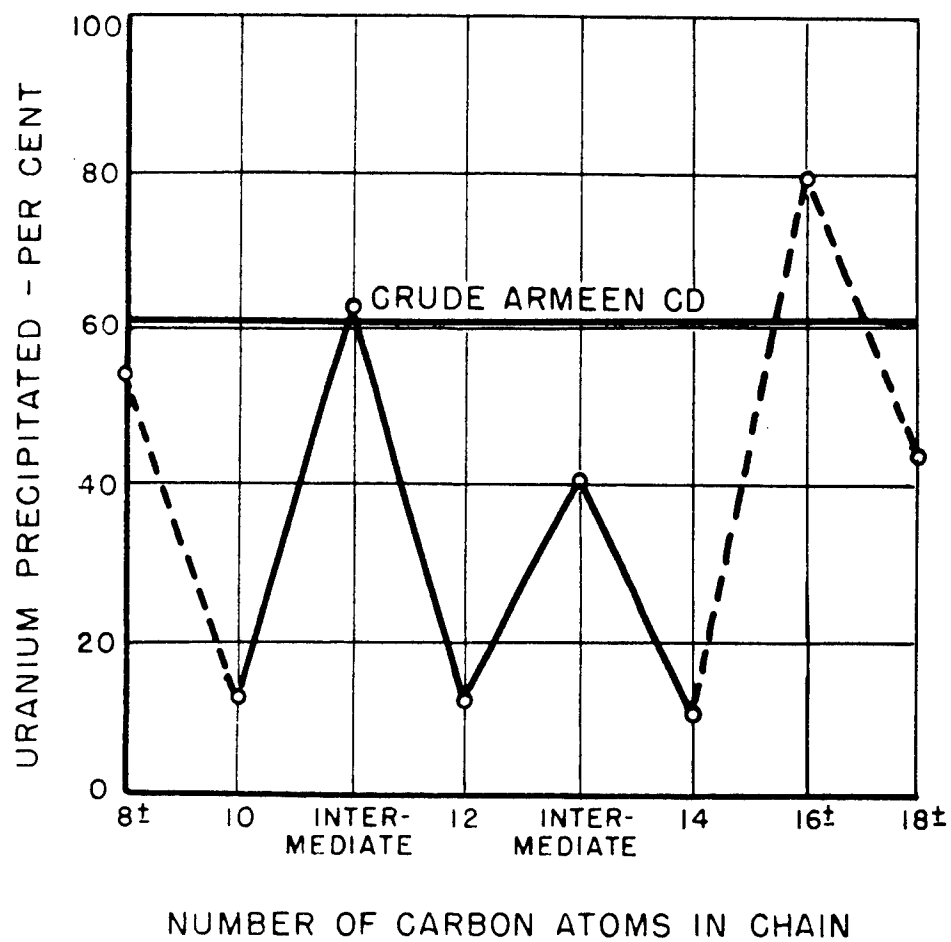


FIGURE 2-PRECIPITATION OF URANIUM FROM  
SUPERPHOSPHATE SOLUTIONS USING  
ARMEEN CD FRACTIONS

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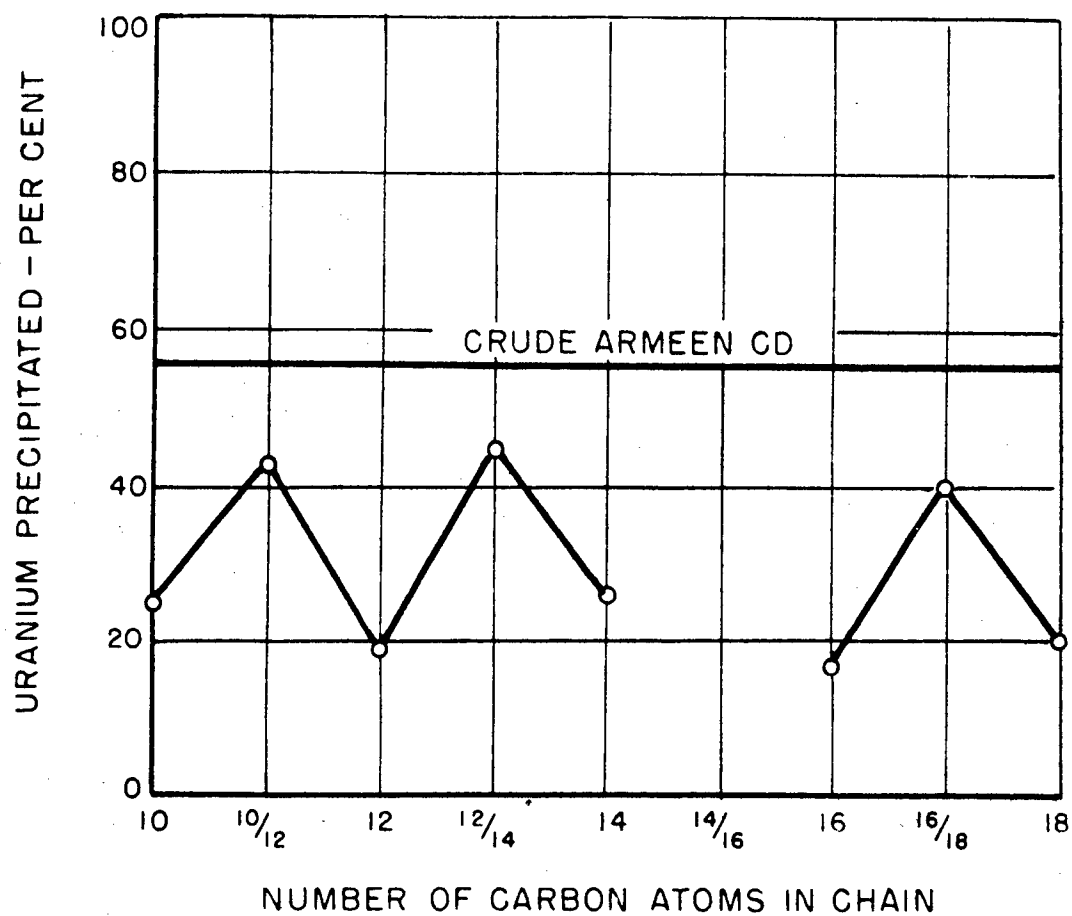


FIGURE 3-PRECIPITATE OF URANIUM FROM SUPERPHOSPHATE SOLUTIONS USING EVEN CARBON CHAIN AMINES

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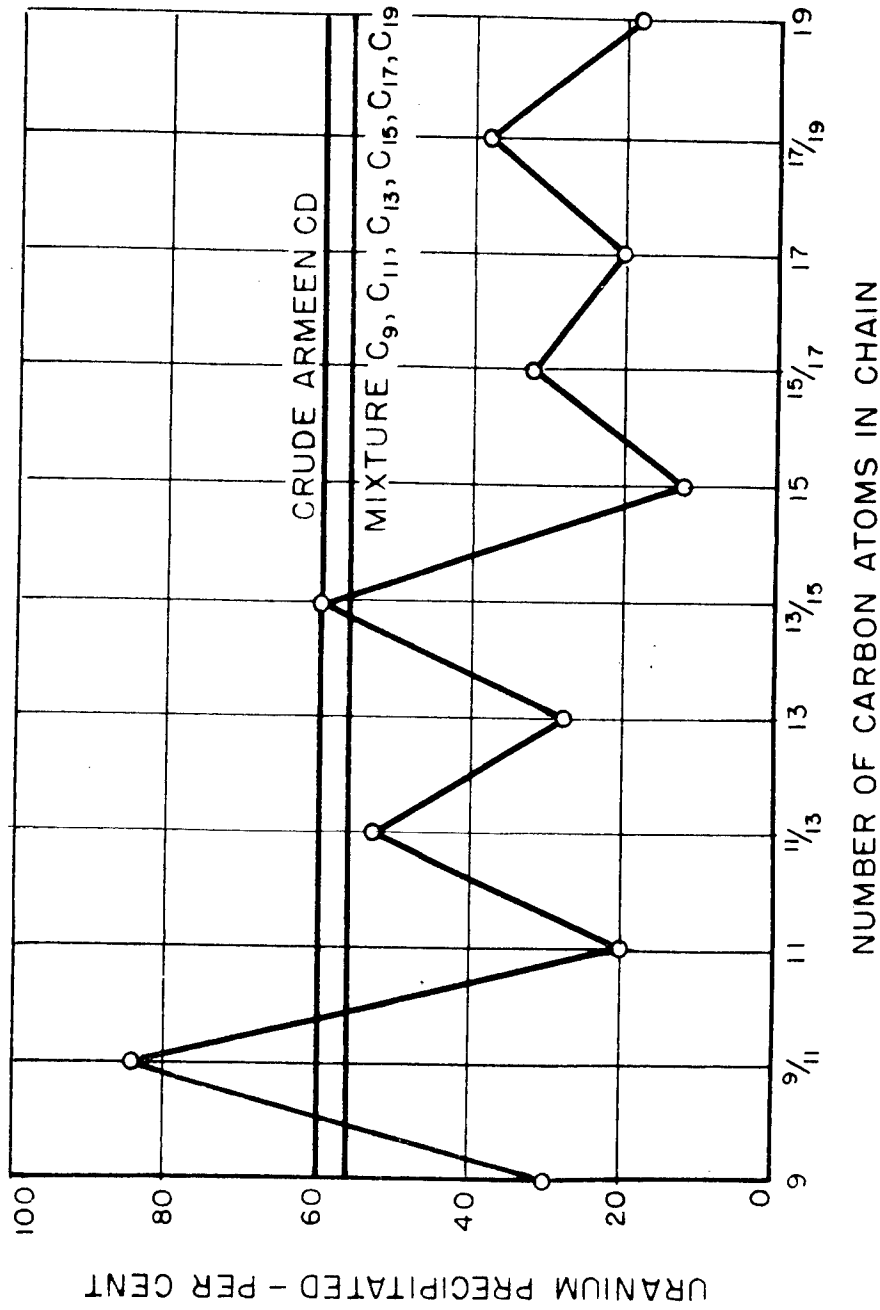


FIGURE 4-PRECIPITATION OF URANIUM FROM SUPERPHOSPHATE SOLUTIONS  
USING ODD CARBON CHAIN AMINES

0-8688